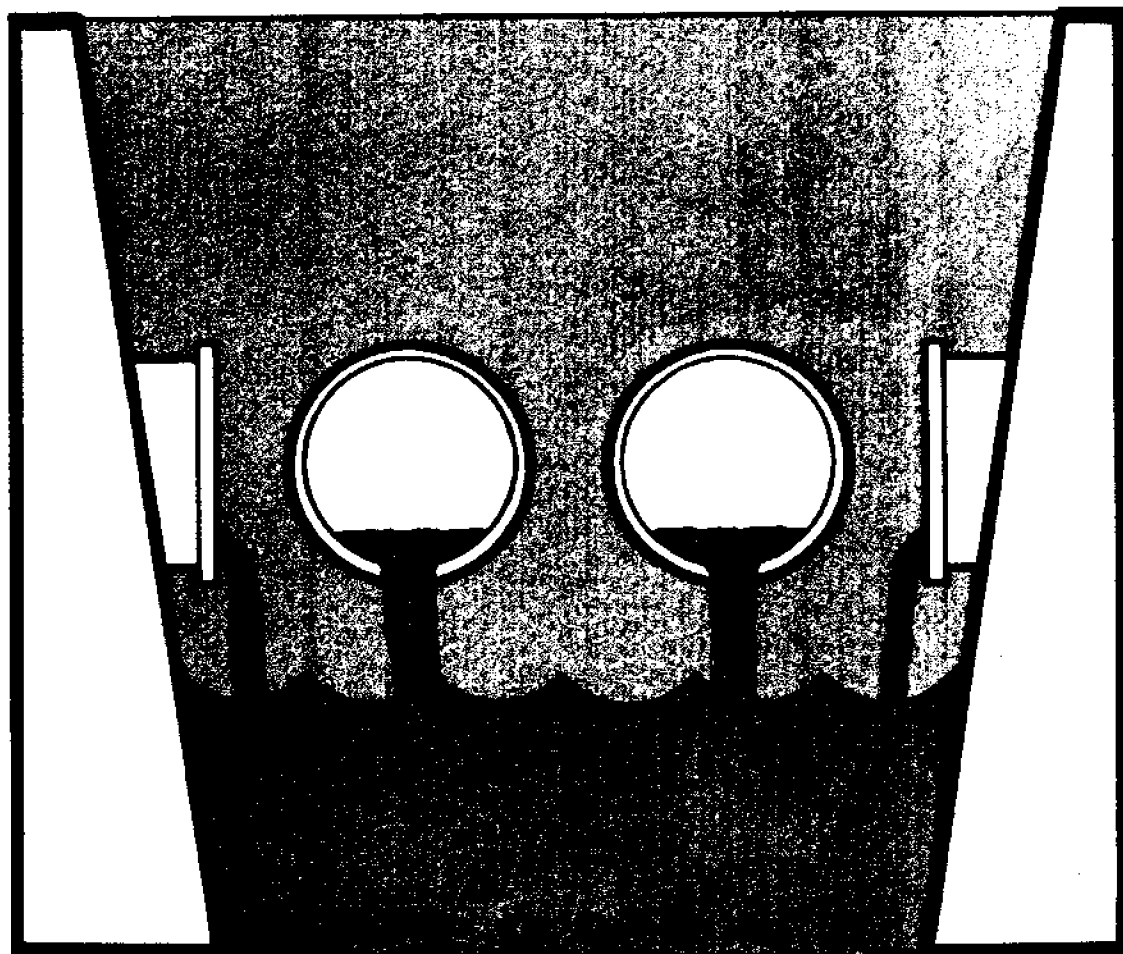


Trace Metal Baseline Studies

on the Murderkill & St. Jones Rivers, Delaware Coastal Plain

Frederick Bopp III, Frederick K. Lepple & Robert B. Biggs

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**This work is a result of research sponsored by NOAA Office of Sea Grant, Dept. of Commerce, under Grant no. 2-35223
college of marine studies university of delaware newark, delaware 19711**

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TRACE METAL BASELINE STUDIES
ON THE MURDERKILL AND ST. JONES RIVERS
DELAWARE COASTAL PLAIN

Frederick Bopp III
Graduate Student
Department of Geology

Frederick K. Lepple
Graduate Student
College of Marine Studies

and

Robert B. Biggs
Associate Professor of Geology
College of Marine Studies

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College of Marine Studies
University of Delaware
Newark, Delaware
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PREFACE

This report represents an interim record of progress during Fiscal Year 1972 in one aspect of the geological investigation of Delaware Bay. Preliminary interpretations have been included.

The authors felt that the inclusion of a layman's definition of "part per million" would be beneficial in order to emphasize to all readers the magnitude of the numbers with which we deal in trace metals. Dale W. Jenkins, director of the ecology program in the Office of Environmental Sciences of the Smithsonian Institute contributed this definition. "The world's driest martini: one ppm of vermouth would be the equivalent of one ounce of vermouth in 7,800 gallons of gin." (Science, 177: 476-77, 1972)

We thank Holly Bopp for her assistance in doing the major sampling efforts for this study, and William Treasure of the Department of Geology for his assistance in doing the remainder.

The National Science Foundation, the National Oceanic and Atmospheric Administration, and agencies of the State of Delaware are free to use the contents in any way which serves the public interest, but are requested to respect the intention of the authors to publish the formal results of their investigation at a later date.

BACKGROUND

Numerous studies of the Murderkill River have been instituted in the past few years since the decision to construct a regional sewage treatment facility for Kent County, Delaware. This new facility, with a design capacity of approximately 10 million gallons per day (which will account for up to 37% of the fresh water daily volume during periods of low runoff), will deposit its 48-hour, extended aeration treated effluent into the Murderkill River approximately 7.3 miles upstream from the river mouth at Bowers, Delaware. The studies done have concerned themselves mainly with the hydrography, biochemistry and ecological balance of the estuary in its present state (see, for example: Daiber, 1972; DeMichele, 1972). The DeMichele study attempted to model the estuary and predict the long-range effects of the sewage treatment facility on the Murderkill River. None of the studies encountered by these authors, however, has established a baseline of the trace metals levels in the riverbed of the as yet virtually unpolluted Murderkill. In view of the significant amounts of lead, copper, chromium, zinc and other metals known to be in sewage treatment wastes, it would seem that the establishment of such a baseline datum is needed in order to make a complete background study of the river in its present condition.

INTRODUCTION

It is the principal purpose of this study to establish a trace

metal baseline for the Murderkill River prior to commencement of operation of the Kent County Regional Sewage Treatment Plant, presently under construction near Frederica, Delaware. Baselines will be established for iron, magnesium, zinc, chromium, copper, lead, cadmium, mercury, nickel and strontium. In addition to the establishment of these baselines, the second purpose of this study is to contrast the Murderkill River baseline with a similarly established baseline for the St. Jones River, which has been the recipient of domestic sewage and industrial and other organ wastes for a number of years. By comparing the results of these two river systems' baselines, it is hoped that some projection may be made of those metals which should be reasonably closely monitored after commencement of operation of the sewage treatment facility.

PHILOSOPHY, AND THE CONCEPT OF ENVIRONMENTAL ACTIVITY

The basic philosophy subscribed to in undertaking this research is that the trace metals which are of interest are only those which are available for introduction to the marine food web through naturally occurring biological or chemical processes--i.e., those trace metals which are "environmentally active." Those cations so firmly bonded to-, or exchanged within-, mineral grains that natural biochemical processes cannot remove them are of no concern to this investigation. The laboratory extraction procedure used was designed to approximate, however crudely, the severest naturally occurring biochemical conditions conceivable, without completely degrading the

sediments. It must be borne in mind, however, that any given sediment particle enters our laboratory extraction system only once, and then is gone from consideration: in natural systems the epi- and infauna may have more than one opportunity to strip cations from any given sediment particle. This is partial justification for making the treatment used here as severe as it is. In addition to this is the fact that the extraction technique used here is a simple, reproducible, inorganic process, whereas the gut-chemistry of even the simplest biological specimen is a complex scheme of enzymes, catalysts and organic acids. The results of the inorganic technique used here are generalized and have a wide range of applications, whereas the results of a biochemical extraction would be species-specific and, therefore, of limited use.

For the purpose of this research the environmentally active trace metals are defined to be those cations which can be separated from 3 grams of dried and disaggregated sediment, from the silt and clay fraction, by leaching with 500 mls. of 10% (vol/vol) hydrochloric acid in distilled/deionized water, at 70°C for 96 hours.

SAMPLING

All samples for this research were taken from a small outboard runabout, R/V Ariadne, with a modified Forster-Anchor Dredge (see Kraft, 1971). Samples were taken at approximately every half- to three-quarters of a mile from the mouth of the rivers to well above the limit of salt intrusion on all branches of the rivers. The

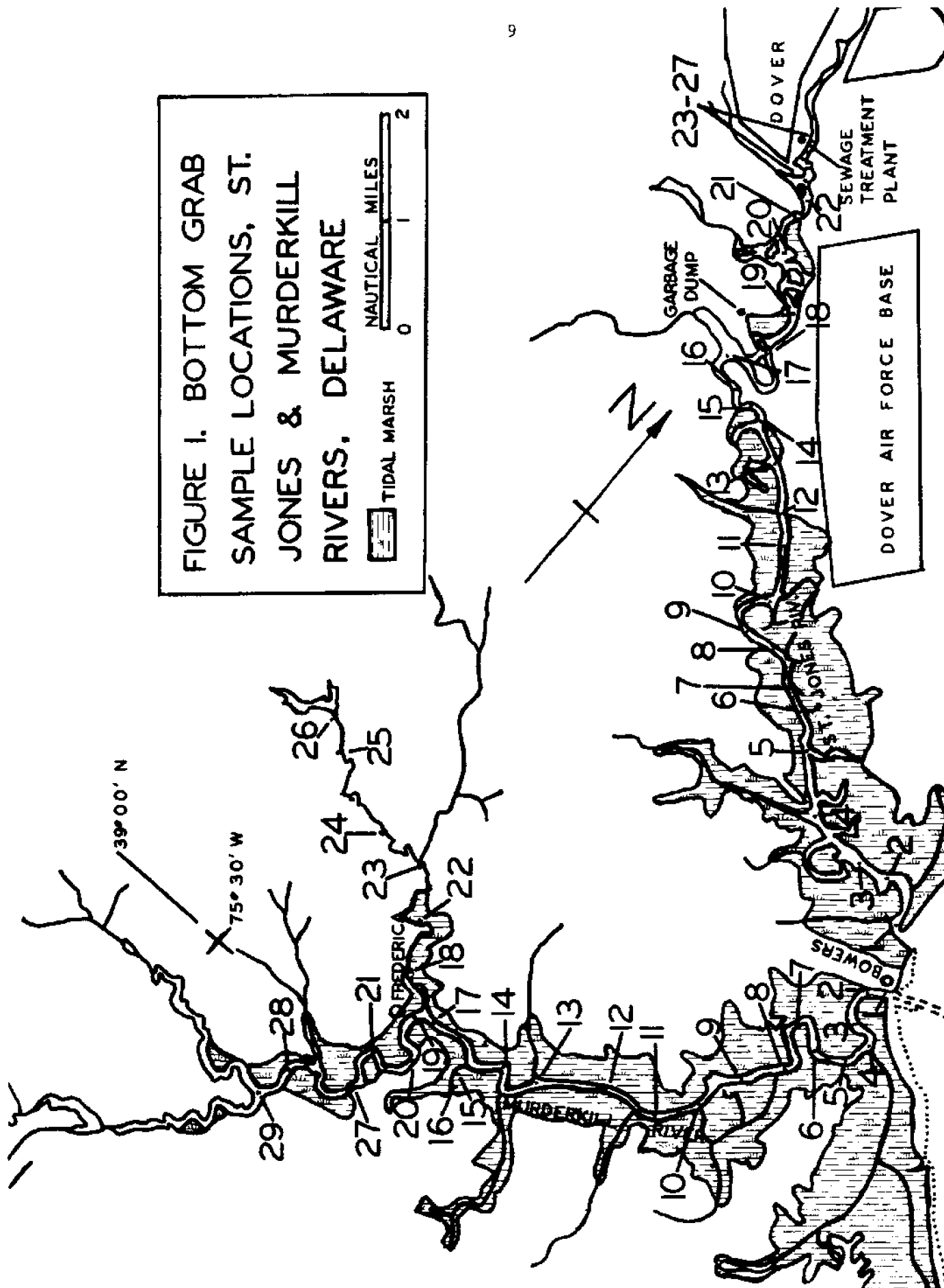
Forster-Anchor Dredge was washed with river water at the sampling site, dropped overboard with approximately 30 feet of line attached, and pulled back aboard manually with a thin veneer of sediments (approximately the top 5 to 10 cm) inside it. These sediments were transferred to heavy gauge plastic sample bags, sealed, and kept cool. Upon return to the laboratory the samples were frozen. Figure 1 is a map of the Murderkill and St. Jones Rivers showing the locations of the samples for each river, along with some of the salient features and landmarks. Sample numbers are preceded by "M" for the Murderkill River, and by "SJ" for the St. Jones River in order to differentiate similarly numbered samples.

LABORATORY PROCEDURES

General: All solutions used in handling, separating, extracting and analyzing the samples were prepared using distilled/deionized water, or Fisher Certified A.C.S. solvents. With the exception of a 3-inch, 63-micron sieve, all laboratory equipment and utensils used in the handling of the samples were made of polyethylene, or other plastic, or of ceramic, in order to eliminate, insofar as is economically practical, the probability of outside contamination. All chemicals used in the analysis were Fisher Certified A.C.S. Reagents, and all standard solutions were prepared from Fisher Certified Atomic Absorption Standards.

Silt and Clay Separation: A subsample of each of the samples, wet weight approximately 250 grams, was transferred to an acid-

FIGURE 1. BOTTOM GRAB
SAMPLE LOCATIONS, ST.
JONES & MURDERKILL
RIVERS, DELAWARE



washed, double AA-water-washed, 600 ml. plastic beaker. ("AA-water" is used interchangeably with "distilled/deionized water.") AA-water was added and the sample aggitated until suspended. The suspensate was passed through a U. S. Standard No. 230, 63-micron mesh sieve, and was collected in an acid-washed, double AA-water washed, 1 liter polyethylene bottle. Then the collected suspensate was centrifuged at maximum allowable RPM in an International Model K Centrifuge for 10 minutes. The supernatant, containing some non-separable colloids, was discarded. The sediment particles were transferred to a 50 ml. plastic beaker and dried at 70°C. The dried sediment was milled to uniform size, determined only by visual approximation, in a Spex Industries Model 8000 Mixer Mill, using a ceramic powder vial and ball. The resulting powder was transferred to a plastic vial, capped and stored.

Trace Metal Extraction: Polyethylene 500 ml. bottles were pre-treated by leaching with 10% (vol/vol) HCl at 70°C for 96 hours immediately prior to use for trace metal extractions. Subsamples of the dried and disaggregated silts and clays were weighed out at 3.00 ± 0.001 grams using a Mettler Analytical Balance. The weighed samples were transferred to the acid-treated bottles and 500 mls. of 10% (vol/vol) HCl were added. The acid was prepared by diluting 50 mls. of concentrated HCl (sp. gr. = 1.19) to 500 mls. with AA-water. The bottles were capped tightly, shaken vigorously, and heated at $70 \pm 4^\circ\text{C}$ for 96 hours. The bottles were shaken and vented periodically during the course of the heated extraction.

When 96 hours had elapsed the solutions were vacuum filtered while hot using a Millipore filtration apparatus with AAWP 0.8 micron filters. The supernatant was returned to the washed bottle in which it had been extracted, capped and stored in a cool place pending analysis.

ANALYTICAL CONDITIONS

All analyses, except those for mercury, were conducted using a Jarrell-Ash Model 800 Atomic Absorption Spectrophotometer in association with twin Honeywell Electronik 17 single-event pen recorders set for 15-inches per hour chart speed. Response of the recorders was 1 second for 1 millivolt full-scale deflection. Hollow cathode lamps used for atomic absorption were all single-element, high spectral output, Jarrell-Ash lamps. The extraction technique used was designed to yield trace metal concentrations within the direct-reading limits of the spectrophotometer for most of the metals of interest. Background corrections were investigated on a routine basis, but no significant differences were noted between corrected and uncorrected readings. As the use of the background correction, B/A mode, reduced the signal-to-noise ratio, it was not used. For specific spectrophotometer operational parameters as applied to individual metals analyses, the reader is referred to Bopp and Biggs (1972).

Mercury was measured by flameless atomic absorption spectrophotometry using a Coleman Model MAS-50 Mercury Analyzer System and

Coleman "Mercury-Free" Reagents. Standards were prepared daily from a stock solution of $1\mu\text{g/ml}$ mercury which had been stabilized in an aqueous acid permanganate solution. The chemistry of the mercury determination is based on the method developed by Hatch and Ott (1968), which measures total mercury (inorganic plus organic forms). However, in our acid treatment, HCl was substituted in place of HNO_3 and H_2SO_4 and no KMnO_4 was added to the leachate. For this reason, oxidation of organic compounds present in the HCl leachate might be incomplete and thus the values reported here are minimum mercury concentrations.

In the analysis procedure, stannous chloride (5ml of 10% solution) was added to 100ml of leachate in order to reduce all of the dissolved mercury to the metallic form. The mercury is then vaporized and circulated by the bubbler system through an absorption cell. The 2537 nm mercury spectral line emitted by a mercury lamp is absorbed by the vapor and the change in transmittance is detected by the phototube. Over the concentration range of 0 to 10ppb, the limit of detection is approximately 0.01ppb mercury.

SOURCES OF ERROR

With any tedious and repetitive laboratory process there is always ample opportunity for contamination of a sample or two by simple human error. The errors which are most costly, however, are those arising from faulty design of the test procedures. In the procedures used here there are at least five places where significant

error may be introduced, either in the form of contaminants or in analytical error. The four major sources of consistent contamination probability are as follow: 1) use of distilled/deionized water which has been contaminated or improperly distilled; 2) contamination due to contact of the sample with the metal sieve during sample preparation; 3) loss of metals due to the discarding of the colloid-containing supernatant after centrifuging; and 4) contamination from the Millipore filter during the filtration of the hot extraction solutions. Each of these four sources was tested for detectable contaminants.

Five hundred ml aliquots of the solutions from each of the possible contaminating steps were measured into 1 liter volumetric flasks. The pH was adjusted to 2.8 by the addition of Bromphenol Blue indicator and the dropwise addition of concentrated HCl. A chelation-extraction yielded a fifty-fold concentration of the metals present due to contamination. Ten ml of 1% Ammonium Pyrrolidine Dithiocarbamate (APDC) were added, shaken vigorously and allowed to react for 15 minutes. The metal-APDC chelates were extracted with 10 ml of Methyl Isobutyl Ketone (MIBK) and the ketone layer aspirated directly into the flame of the spectrophotometer. Several of the metals with high concentrations in the bay were analyzed for--iron, magnesium, zinc, nickel--with negative results in all but the colloid-discarding sample. However, the level noted was not inordinately high--on the order of several hundred ppb iron, several tens of ppb magnesium, traces of zinc and no nickel. Considering that the sample

preparation was carried out on samples still containing bay water, these metal levels probably represent ions from the bay pore waters taken into solution during sample preparation.

A 1 liter aliquot of the colloid-discarding supernatant solution was vacuum filtered using a Millipore filtration apparatus with an HAWP 0.45 micron filter. The retained particulate material weighed 0.0017 grams, or 0.006% of the nominal yield of 30 grams of fine grained material from the sample preparation procedure previously described. This is well below the 0.03% error already found acceptable in the extraction technique previously described, and, therefore, is not considered to be an important source of error.

The last source of error is the machine error caused by resolution limitations inherent in the design of the atomic absorption spectrophotometer. Listed in Table 1, below, is the sensitivity, or resolution limit, of the machine for each metal run. In converting the concentration readings determined for the extraction solutions into concentrations in the finer than 63-micron fraction, the conversion factor of 1/3 was used. Table 1 is a tabulation of the manufacturer's specifications for sensitivity. These specifications were met for all metals except Fe and Mg, since all readings were made by direct determination.

Table 1: Sensitivities for the Data Presented
in Tables 2 and 3, Expressed as - Parts Per
Million of the <63 Micron Sediment Fraction.

Metal	Sensitivity
Fe	0.05 ppm
Mg	0.004 ppm
Zn	0.015 ppm
Cr	0.06 ppm
Cu	0.04 ppm
Pb	0.30 ppm
Cd	0.02 ppm
Hg	0.01 ppb
Ni	0.10 ppm
Sr	0.05 ppm

RESULTS

Tables 2 and 3 contain tabulations of all of the trace metal concentrations determined during Fiscal Year 1972 research. These concentrations are expressed as micrograms per gram, or parts per million, of the sediment fraction finer than 63 microns.

The values presented in Tables 2 and 3 were used to plot the graphs presented as Figures 2 through 11, which illustrate the variations among the trace metals along the length of the riverbeds.

DISCUSSION

From Figures 2 through 11 several patterns make themselves apparent. These patterns appear to assist in the understanding of the sources and distribution of the various pollutive trace metals.

Table 2: Concentrations of Trace Metals in the Murderkill River Bottom Sediments, Expressed as Parts Per Million of the <63-Micron Sediment Fraction. (Mercury Expressed as Parts Per Billion)

SAMPLE	Fe	Mg	Zn	Cr	Cu	Pb	Cd	Hg	Ni	Sr
M1	36250	5700	362	128	35	355	5.7	711	654	206
M2	44850	7250	405	130	50	217	8.7	984	680	275
M3	35600	7500	278	92	50	92	2.7	332	425	123
M4	54300	7400	219	138	26	95	2.8	198	733	134
M5	34000	7350	100	128	38	75	2.7	87	395	72
M6	35500	7850	110	128	38	55	4.0	70	412	58
M7	42150	8000	220	128	22	92	5.0	205	408	60
M8	40350	7850	190	128	22	55	3.7	195	470	73
M9	33150	7100	97	92	22	45	4.0	77	533	73
M10	38350	6500	262	120	33	75	4.0	317	361	75
M11	24500	5150	77	83	30	55	4.5	88	250	78
M12	39150	6800	313	107	50	83	2.7	318	478	72
M13	29000	7000	90	83	12	42	1.8	73	245	78
M14	37150	6650	133	128	38	42	1.8	163	408	68
M15	34000	5150	225	113	67	67	3.7	262	433	58
M16	32000	5100	202	92	67	83	3.2	255	325	65
M17	35150	5500	190	92	55	62	2.3	222	328	53
M18	34650	6250	102	113	55	50	1.3	67	550	62
M19	33500	5000	203	100	38	67	1.8	262	378	75
M20	34650	5000	178	90	30	80	2.8	222	435	58
M21	25000	5650	85	92	22	38	1.3	102	258	58
M22	20000	4600	77	60	72	33	1.3	97	250	58
M23	31150	4600	172	83	38	42	1.3	213	300	60
M24	8500	1150	74	59	202	53	1.7	272	195	51
M25	21000	2550	145	113	92	50	1.3	265	400	42
M26	5100	650	48	56	191	51	0.8	183	178	53
M27	28850	4350	170	92	33	42	1.3	242	400	53
M28	19000	2700	104	104	37	74	1.1	261	372	67
M29	26900	3600	150	111	37	37	2.0	198	695	61

Table 3: Concentrations of Trace Metals in the St. Jones River Bottom Sediments, Expressed as Parts Per Million of the <63-Micron Sediment Fraction. (Mercury Expressed as Parts Per Billion)

SAMPLE	Fe	Mg	Zn	Cr	Cu	Pb	Cd	Hg	Ni	Sr
SJ1	23150	3150	88	145	38	67	1.0	233	417	60
SJ2	44350	8350	360	183	55	133	1.3	467	662	122
SJ3	44000	8000	373	162	47	133	1.3	500	662	107
SJ4	44650	8000	358	200	50	87	0.5	383	453	108
SJ5	42500	7500	368	192	42	117	1.0	500	458	105
SJ6	39350	6500	213	162	25	58	1.0	233	292	92
SJ7	41850	7150	267	162	55	100	1.0	350	408	90
SJ8	38650	5500	277	145	62	100	1.0	375	595	93
SJ9	39350	7350	298	173	47	95	0.5	342	642	82
SJ10	40350	7850	335	162	50	117	1.3	355	628	58
SJ11	45850	8500	335	183	38	87	1.3	272	450	50
SJ12	33850	6650	367	157	42	100	2.3	337	317	42
SJ13	34350	5350	255	140	62	95	1.3	300	312	58
SJ14	33150	4350	227	107	38	95	1.8	262	275	63
SJ15	22650	5000	127	80	30	42	1.0	138	208	77
SJ16	30350	4750	310	107	33	125	2.3	347	403	52
SJ17	33150	4850	333	127	68	133	2.3	375	312	52
SJ18	29500	3650	270	87	38	113	2.7	342	350	30
SJ19	25850	3500	347	100	55	133	2.3	450	292	45
SJ20	25850	3600	355	107	72	130	2.3	467	287	60
SJ21	24500	3350	438	100	88	142	2.3	550	320	50
SJ22	24000	3650	505	120	130	142	1.8	750	353	53
SJ23	23850	3250	457	127	92	155	2.7	667	383	58
SJ24	23000	3350	505	133	117	237	2.3	867	312	77
SJ25	21000	4650	833	113	183	350	3.7	2833	308	73
SJ26	25850	3150	740	167	125	583	2.7	1267	342	77
SJ27	25000	5000	467	120	162	687	7.7	3083	353	103

In the graphs illustrating the distribution of strontium, mercury, cadmium, lead and zinc, the samples from the Murderkill River indicate high values in association with Bowers, Delaware. It seems obvious from these high values that the town of Bowers has a significant local pollutive effect upon the river. This effect is definitely local, however, dissipating less than one mile upstream, with the exception of cadmium which appears to be much more mobile than the other metals.

In most cases, there is a subtle but definite gradual lowering of metal levels from rivermouth to headwaters, as one would surmise would be the case with a tidal estuary. This trend would suggest that at least several of the pollutive metals are being transported into the lower reaches of the rivers by the tidal movement of the bay waters. Sea Grant Report No. DEL-SG-9-72 indicated that there is a trace metal "sink" in the vicinity offshore from the mouths of the Murderkill and St. Jones Rivers, so this net inshore and upriver transport of metals by tidal fronts is not entirely unreasonable.

In two cases, the upper reaches of the Murderkill River show unusually high values. In the one case, the Main Channel above Frederica indicates unusually high concentrations of nickel, and in the other case, the Spring Creek Branch indicates unusually high concentrations of copper. It is difficult to associate these anomalies with a direct cause by simple observation. However, in both cases there are farming activities closely associated with the

area sampled, and this might provide a possible explanation.

The most obvious trend one is struck with from Figures 2 through 11 is the extreme trend in the values of trace metals in the uppermost 3 miles of the St. Jones River. These 3 miles are the section of the St. Jones most closely associated with urban activity centered in Dover. Indeed, the last four samples in the series approach the sewage effluent outflow for the treatment plant currently servicing Dover. One can see that there are varying degrees of effect from the urban activities, although, in almost all cases, the effect is one of heightening the trace concentrations in the area. For zinc the heightened concentration is quite obvious: the effect of the urbanity is not necessarily a local one, with higher zinc concentrations noticeable for at least five miles downstream. A similar phenomenon, although not so extreme, is notable for the distribution of chromium. The heightened concentrations are marked for copper, lead, cadmium, and mercury. The downstream effects from the sources of trace metals associated with Dover are such that the general trace metal levels for the St. Jones River are higher than those of the Murderkill River for the following metals: zinc, chromium, copper, lead, and mercury. For these metals, it is significant that if there are common values in both rivers, they occur at 7-8 miles above the rivermouths, the approximate location of Frederica on the Murderkill.

CONCLUSIONS

1) Trace metal baselines have been established for the Murderkill River and the St. Jones River with respect to iron, magnesium, zinc, chromium, copper, lead, cadmium, mercury, nickel and strontium. It is hoped that further work may be accomplished in the foreseeable future which will expand the results of this work.

2) Local pollution sources along the Murderkill River may be identifiable by their associated trace metal accumulations--most notable here is Bowers, Delaware--but the Murderkill River appears to have extremely low trace metal concentrations along most of its course. This bears out the reputation of the river as being virtually unpolluted.

3) The effects of large-scale urban population concentrations can be demonstrated by their associated trace metal concentrations. The large-scale effects of urban trace metal pollution have been demonstrated by the comparison of the baselines of the two rivers.

4) It is recommended that the Kent County Regional Sewage Treatment Plant be monitored for trace metal output, and that the river bottom sediments be similarly monitored in order to trace the growth of any front which may arise due to effluent dumping practices at the new facility.

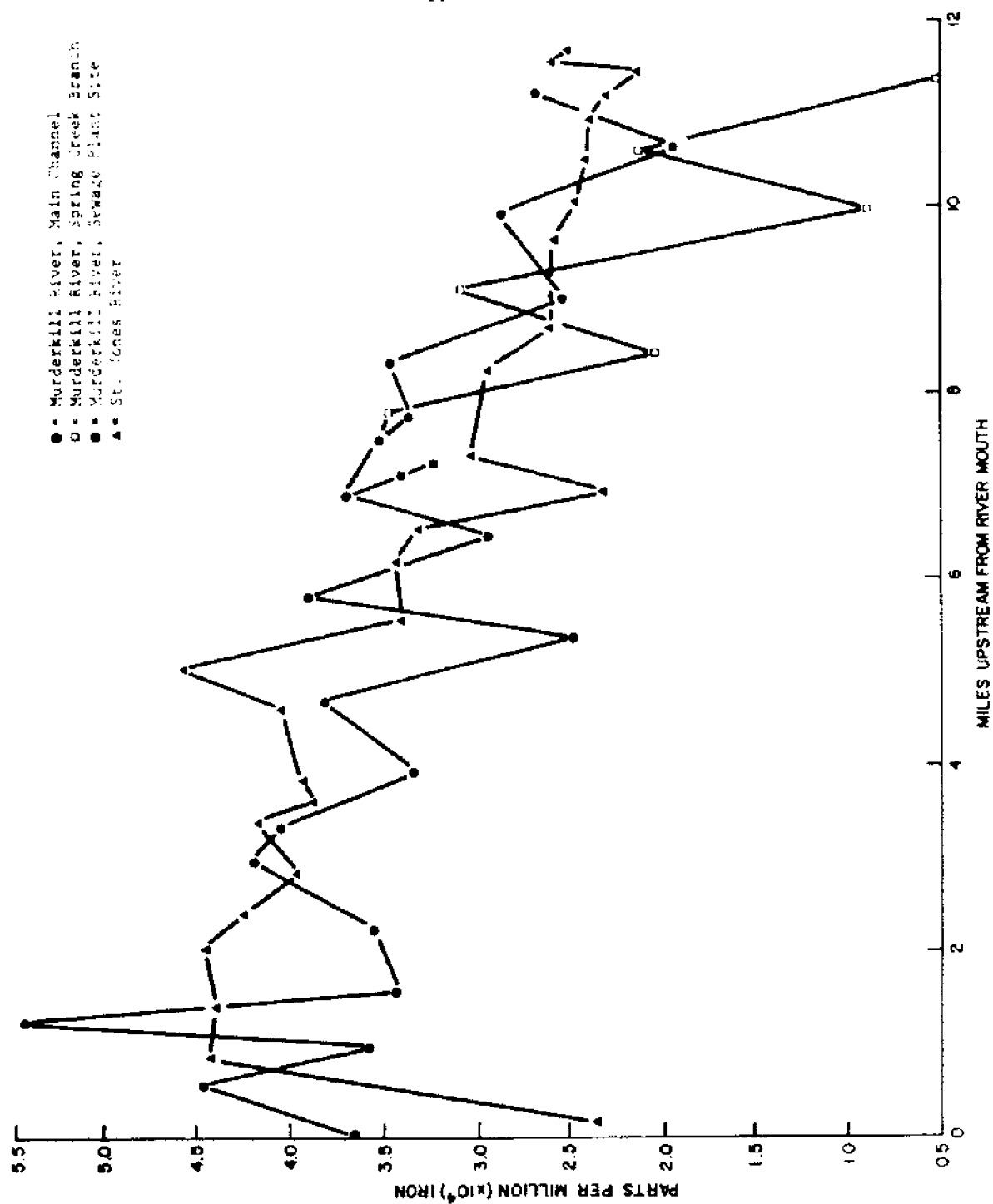


FIGURE 2: Distribution of environmentally Active Fe in Murderkill and St. Jones River Bottom Sediments, Expressed in PPM of the <63 Micron Sediment Fraction.

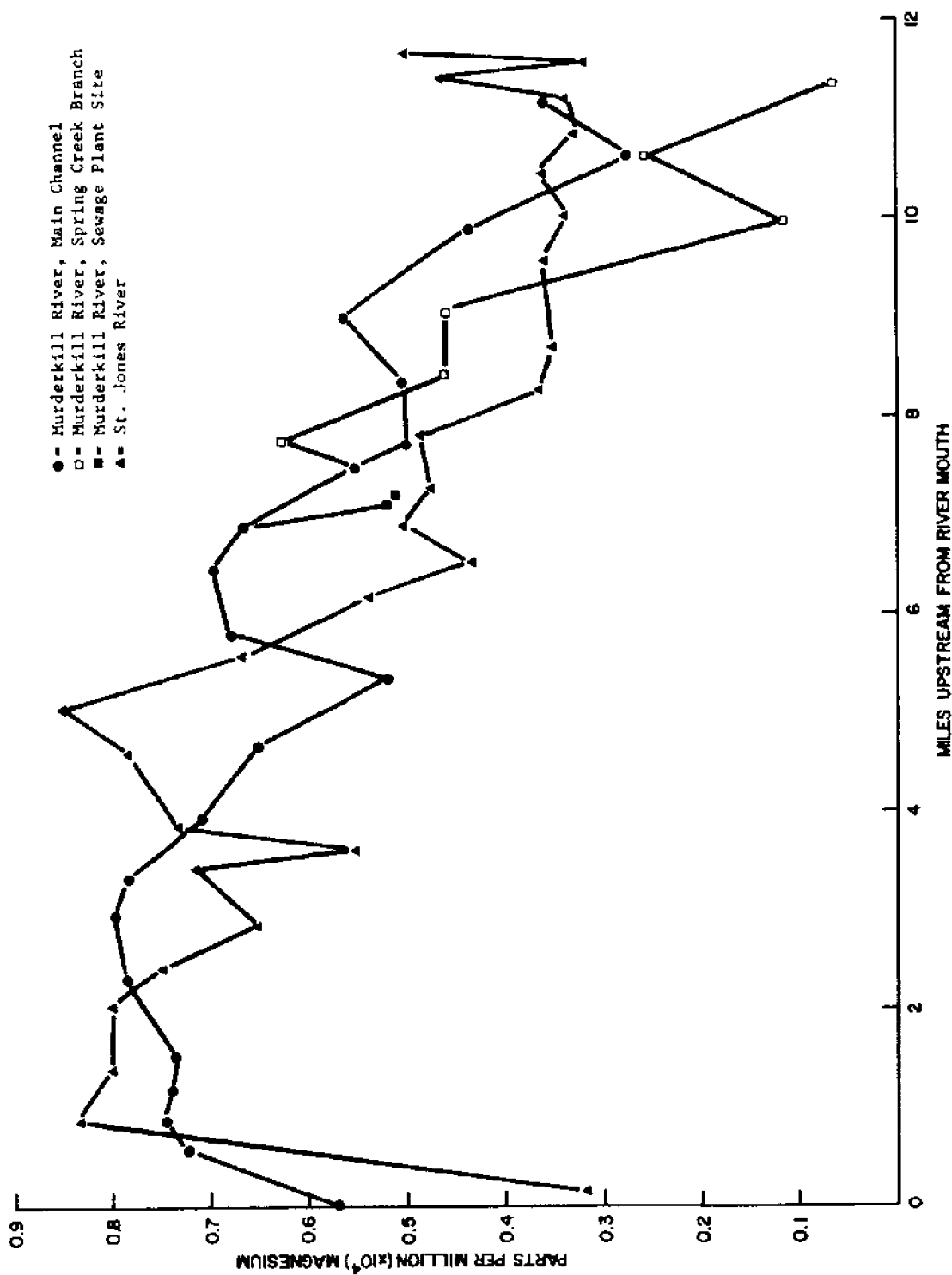


FIGURE 3: Distribution of Environmentally Active Mg in Murderkill and St. Jones River Bottom Sediments, Expressed in PPM of the <63 Micron Sediment Fraction.

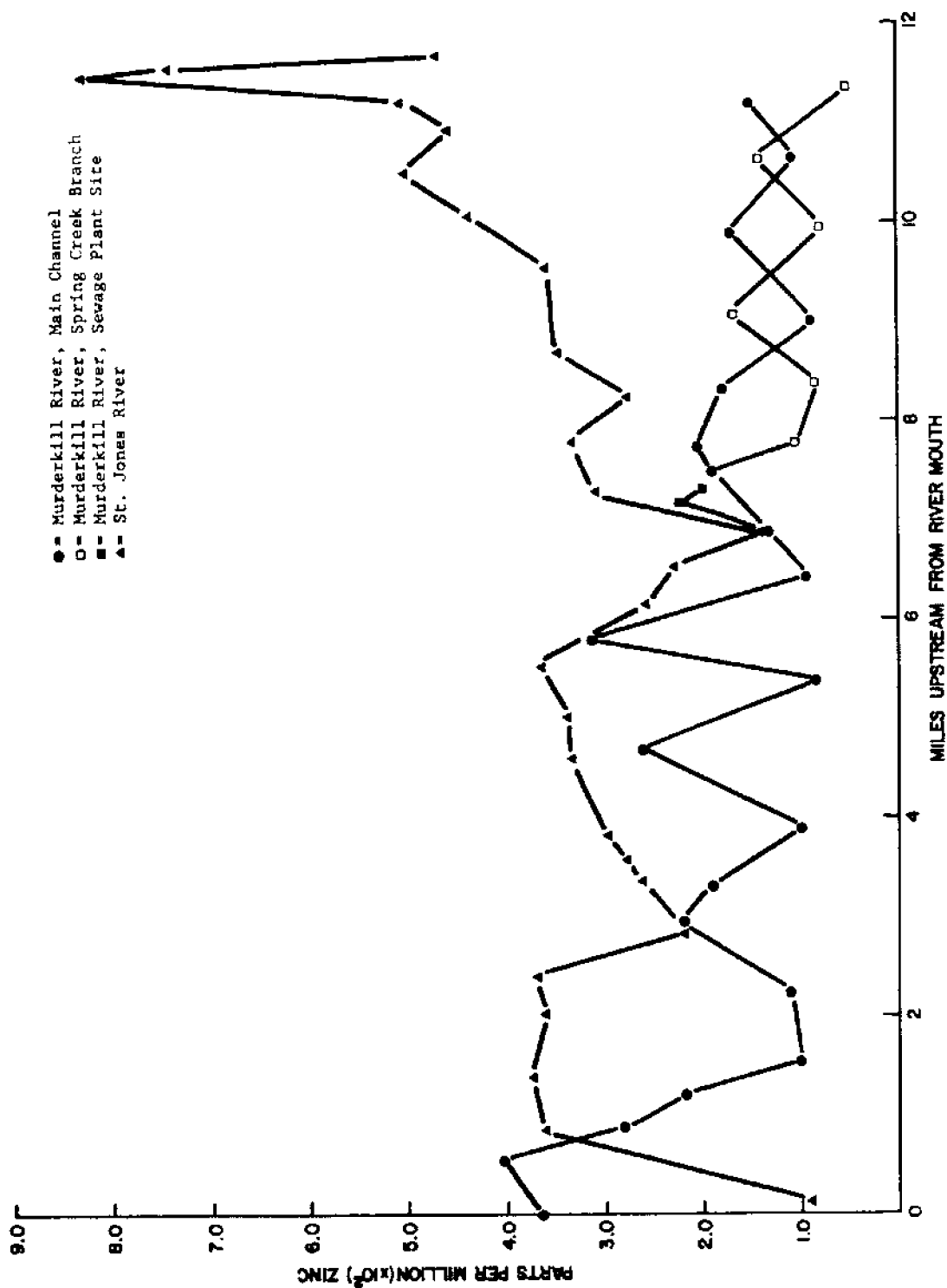


FIGURE 4: Distribution of Environmentally Active Zn in Murderkill and St. Jones River Bottom Sediments, Expressed in PPM of the <63 Micron Sediment Fraction.

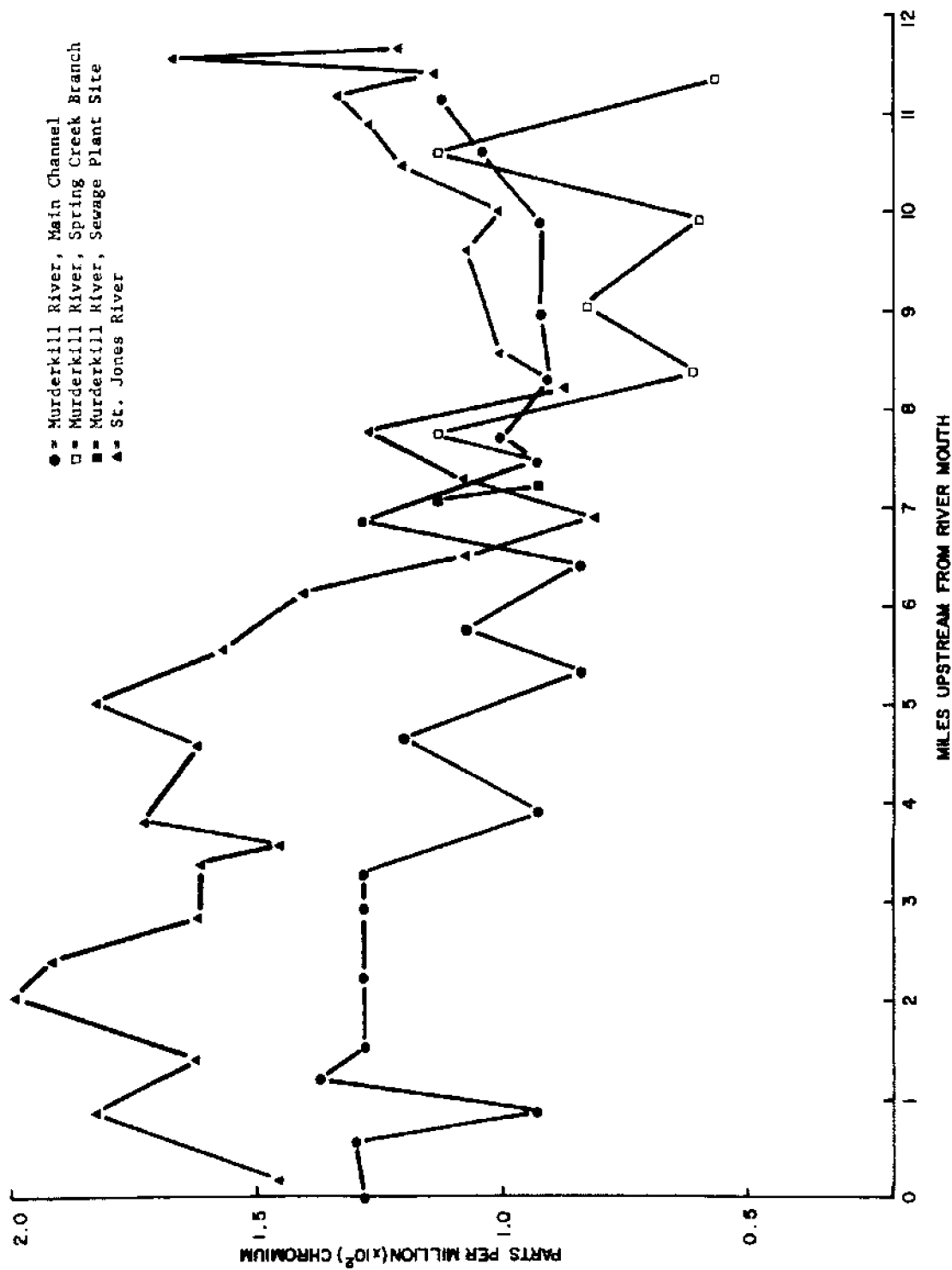


FIGURE 5: Distribution of Environmentally Active Cr in Murderkill and St. Jones River Bottom Sediments, Expressed in PPM of the <63 Micron Sediment Fraction.

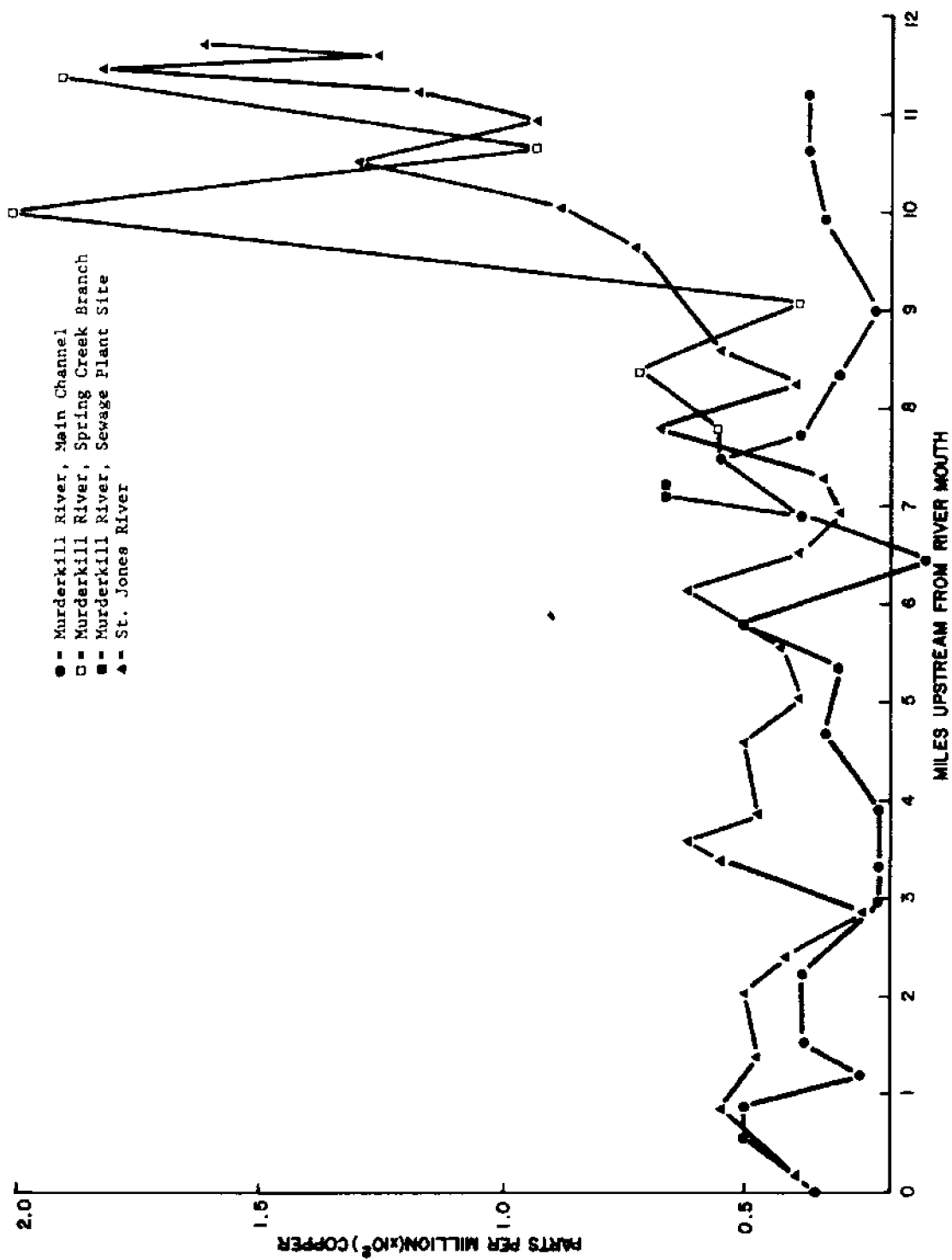


FIGURE 6: Distribution of Environmentally Active Cu in Murderkill and St. Jones River Bottom Sediments, Expressed in PPM of the <63 Micron Sediment Fraction.

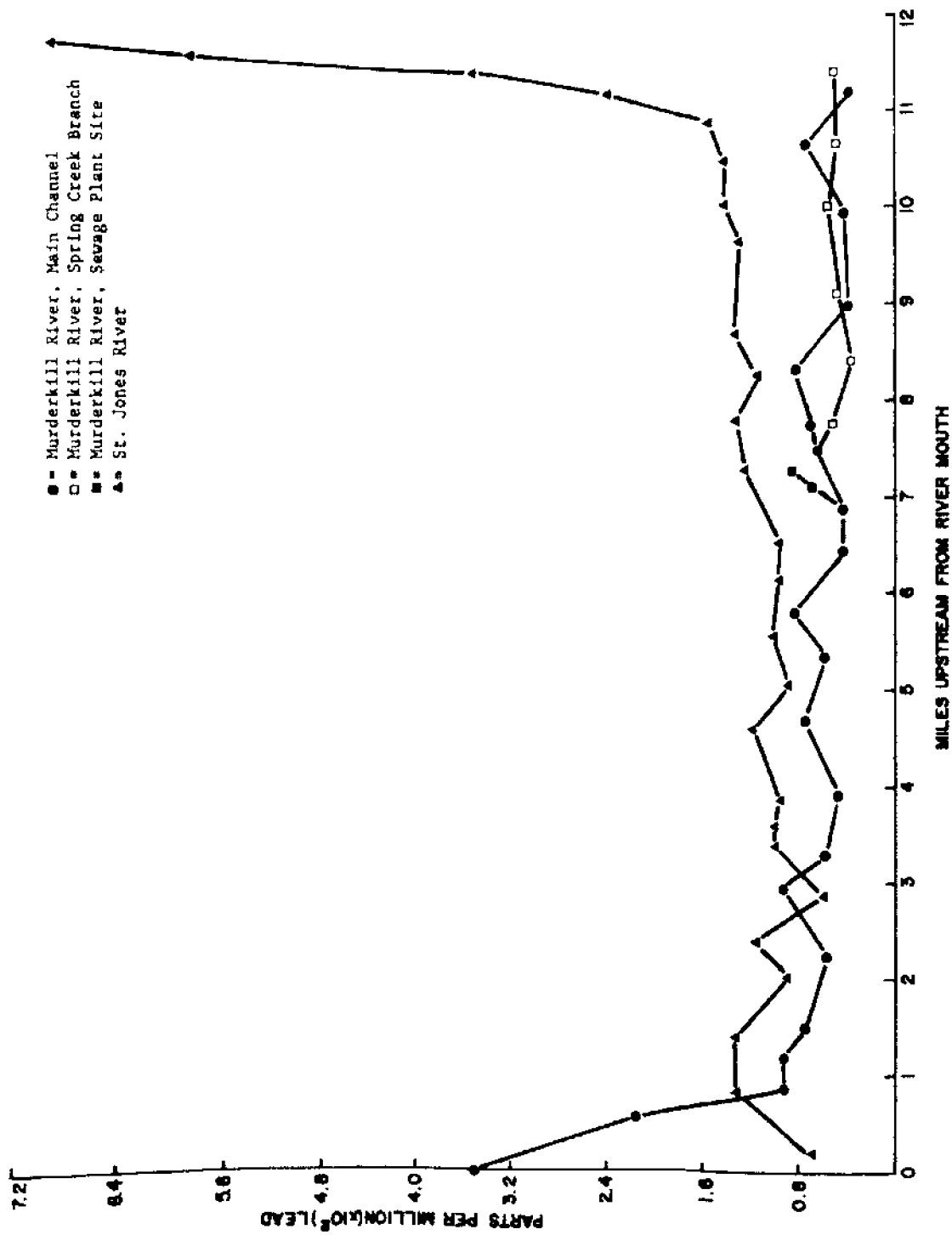


FIGURE 7: Distribution of Environmentally Active Pb in Murderkill and St. Jones River Bottom Sediments, Expressed in PPM of the <63 Micron Sediment Fraction.

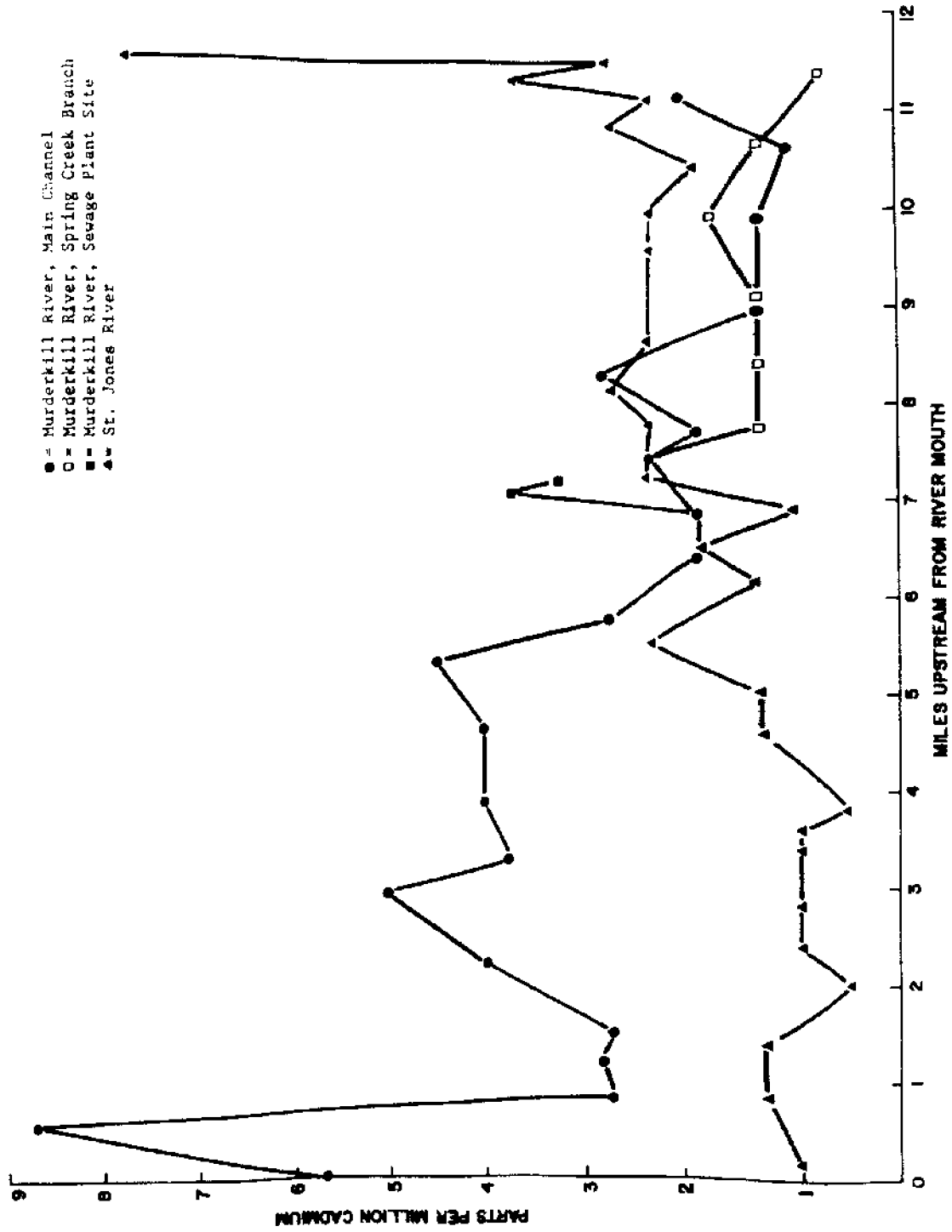


FIGURE 8: Distribution of Environmentally Active Cd in Murderkill and St. Jones River Bottom Sediments, Expressed in PPM of the <63 Micron Sediment Fraction.

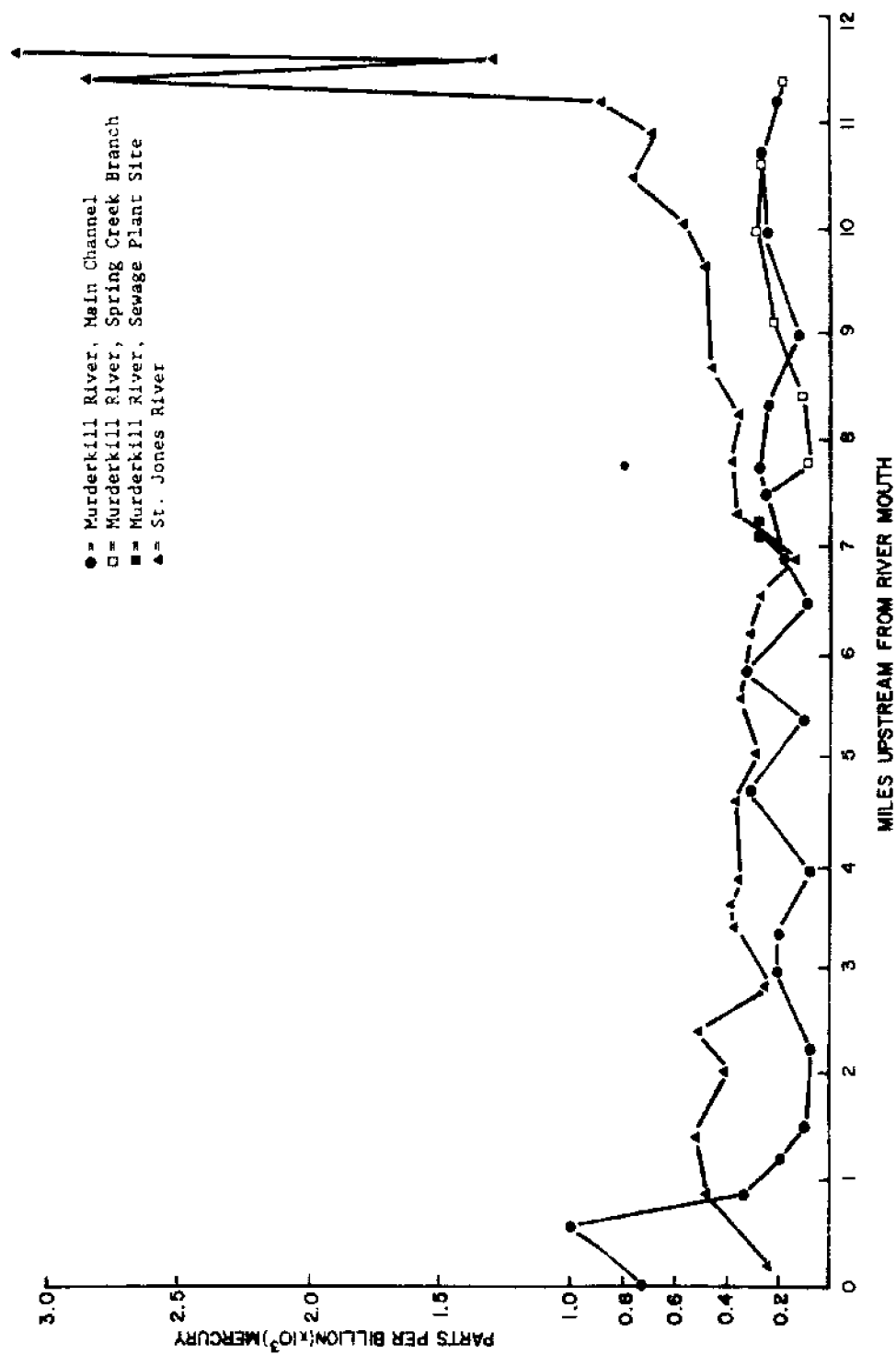


FIGURE 9: Distribution of Environmentally Active Hg in Murderkill and St. Jones River Bottom Sediments, Expressed in PPB of the <63 Micron Sediment Fraction.

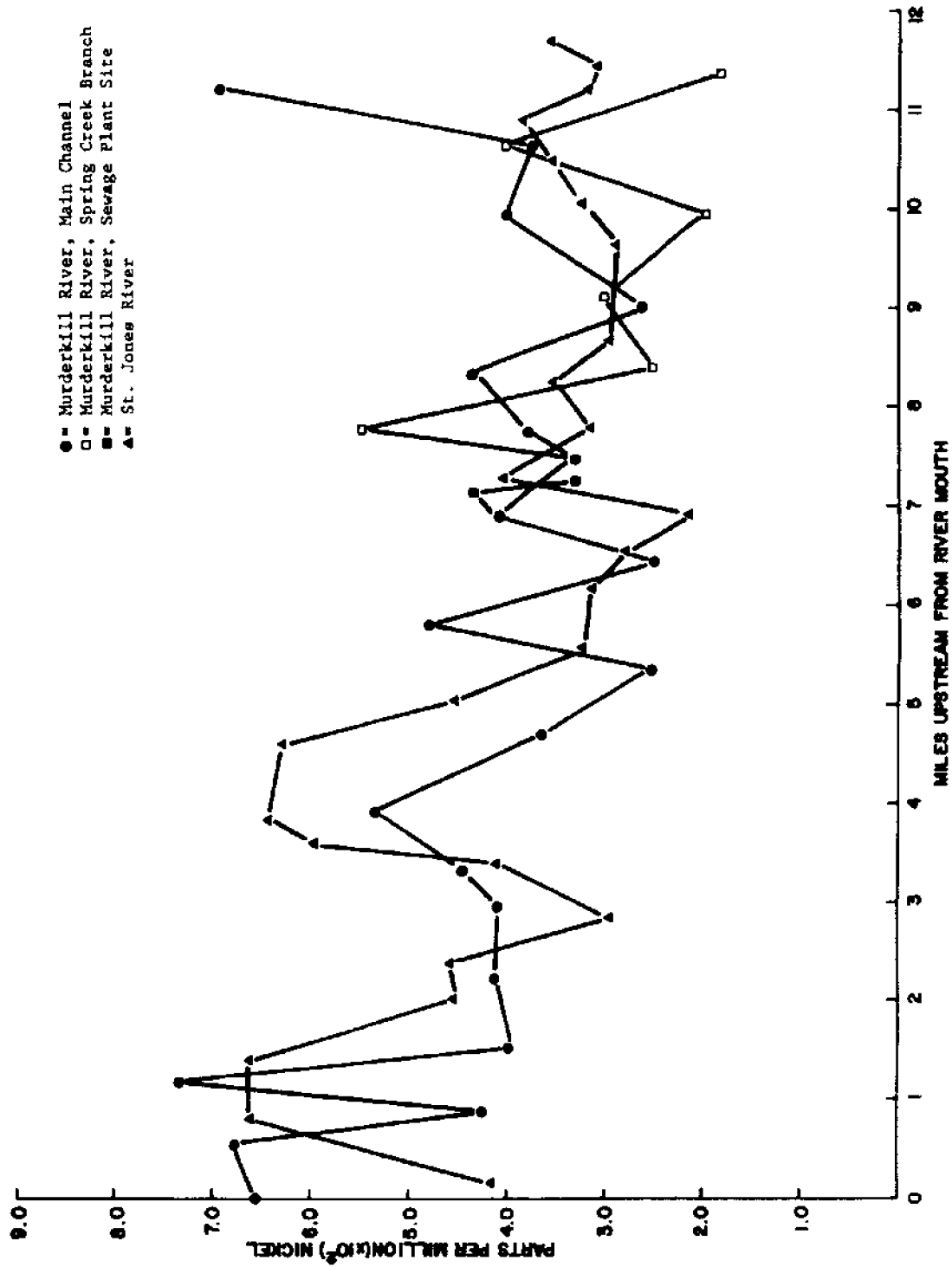


FIGURE 10: Distribution of Environmentally Active Ni in Murderkill and St. Jones River Bottom Sediments, Expressed in PPM of the <63 Micron Sediment Fraction.

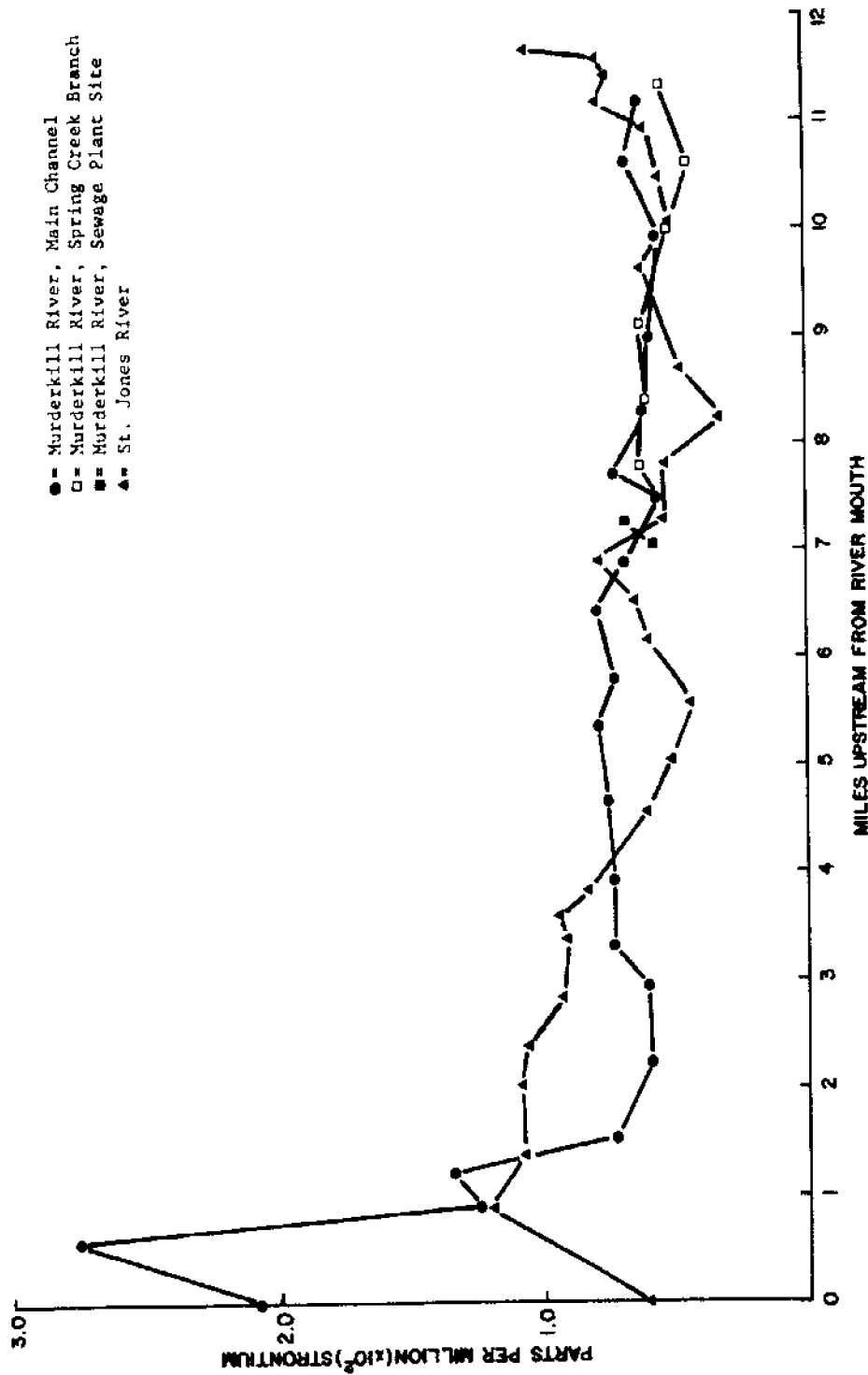


FIGURE 11: Distribution of Environmentally Active Sr in Murderkill and St. Jones River Bottom Sediments, Expressed in PPM of the <63 Micron Sediment Fraction.

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